BAT457

eXtreme Fast Charge Cell Evaluation of Lithium-ion Batteries

# METHODS FOR THE DETECTION AND QUANTIFICATION OF LITHIUM PLATING

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U.S. DEPARTMENT OF ENERGY | Energy Efficiency & Renewable Energy
VEHICLE TECHNOLOGIES OFFICE

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AMR, Arlington, VA

# OVERVIEW

## Timeline

- Start: October 1, 2017
- End: September 30, 2021
- Percent Complete: 75%

## Barriers

- Cell degradation during fast charge
- Low energy density and high cost of fast charge cells

## Budget

- Funding for FY20 – $5.5M

## Partners

- Argonne National Laboratory
- Idaho National Laboratory
- Lawrence Berkeley National Lab
- National Renewable Energy Laboratory
- SLAC National Accelerator Lab
- Oak Ridge National Lab

# Relevance

## Impact

- Decrease charging time without sacrificing lifetime
- Connect Li plating heterogeneity to local heterogeneities
- Mitigate Li plating to reduce capacity fade from extreme fast charging

## Objectives

- Develop approaches to accurately detect and quantify Li deposition during extreme fast charge conditions
- Link detection of onset of Li with cell performance and local heterogeneity

# MILESTONES

## High level Li detection related milestones in XCEL

| Milestone | End Date | Status |
|---|---|---|
| Identify and contrast strengths/weaknesses of global (not localized), nondestructive detection techniques (on CAMP single layer Round 2 pouch cells) | 3/30/2020 | Completed |
| Identify and contrast strengths/weaknesses of localized and/or destructive Li detection techniques. Identify where to combine techniques to span length scales | 9/31/2020 | On Track |
| Combining at least 2 techniques to study when, where, and/or how Li plates on the same electrode<br>• Combine electrochemical & chemical/crystallographic signatures<br>• Combine to quantify techniques to discover detection limits | 9/31/2020 | On Track |
| Link detection of onset of Li with cell performance and other cell/cycling properties (aging); how does that evolve with aging | 9/31/2020 | On Track |

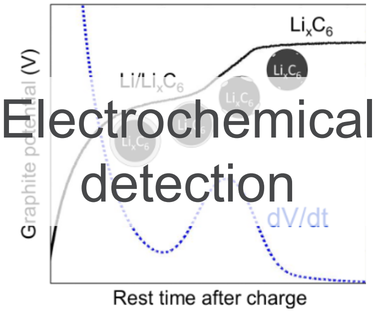


Any proposed future work is subject to change based on funding levels

# Approach

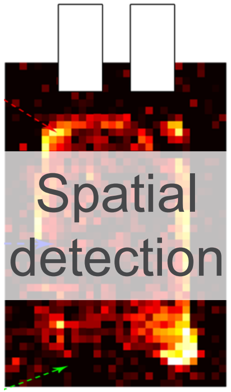
**Explore and develop several complementary techniques to answer:**

- When does Li plating start?
- Where does Li plate first?
- How does Li plating connect to local heterogeneity?
- Which non-destructive, readily accessible technique should we use as a standard for all XFC studies?




Electrochemical detection

Global quantification



Spatial detection

# Electrochemical Detection

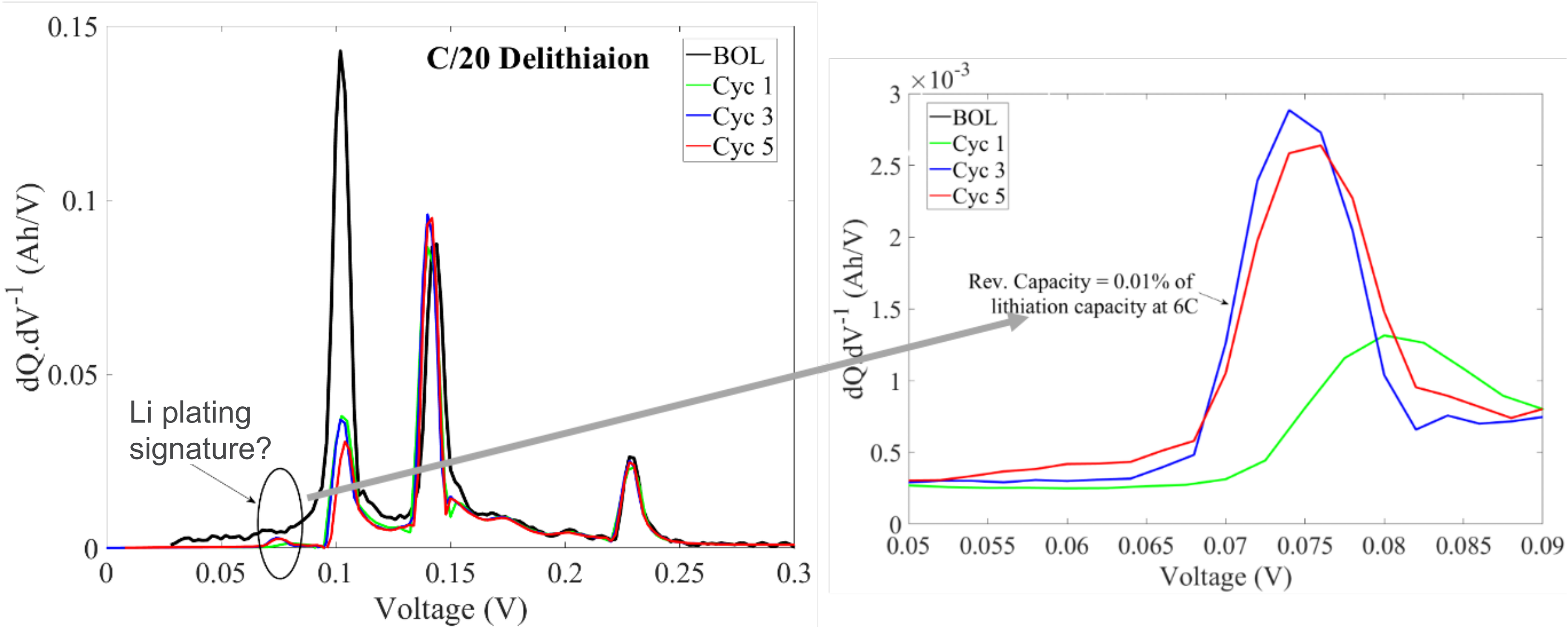
Goal: identify electrochemical signature of Li plating

- Signature to be used as standard detection of Li plating across project

# Finding dQ/dV signature during delithiation

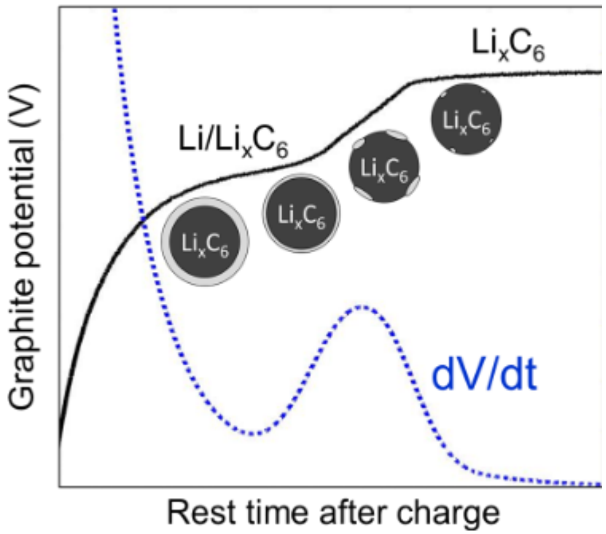
Reversible capacity very small (0.01% of lithiation capacity)

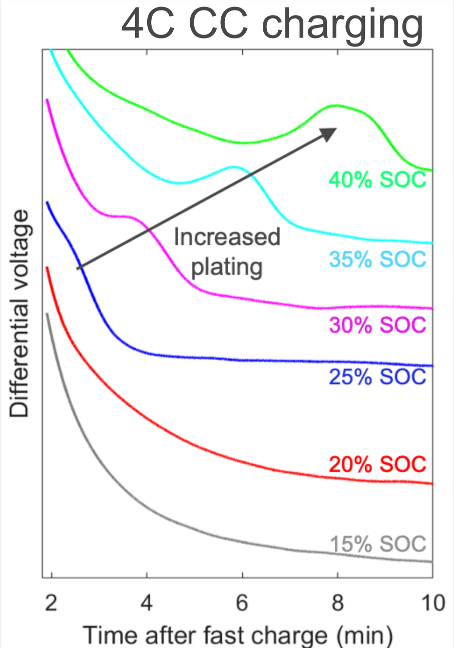
➢ thus very weak dQ/dV signature during slow delithiation

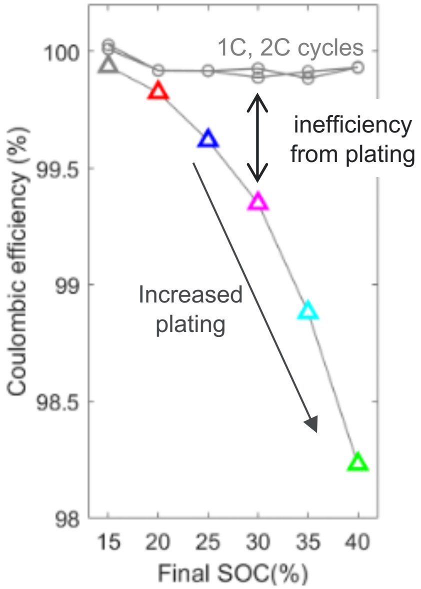



Tanvir Tanim, Parameswara Chinnam, Eric Dufek, Charles Dickerson (INL)

# Observe dV/dt at rest



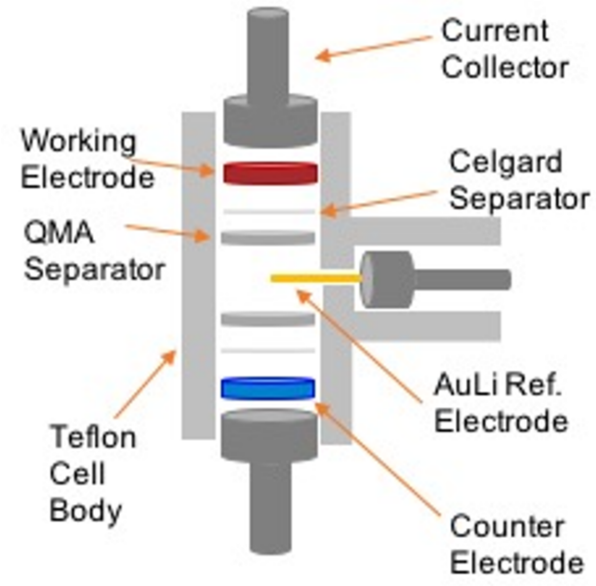




- Chemically intercalation of Li metal during rest gives peak feature in dV/dt (left)
- Li plating observed for CC charging above 25-30% SOC (middle & right)
- Technique sensitivity: ~1% of graphite capacity of plated Li (0.03 mAh Li for 3 mAh electrode)

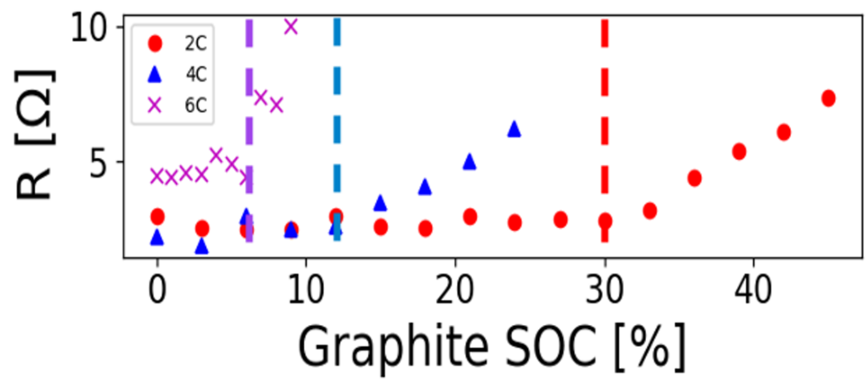



Zach Konz, Eric McShane, David Brown, Bryan McCloskey (Berkeley)

# Distribution of Relaxation Times Electrochemical Impedance Spectroscopy (DRT-EIS)




- Detect Li plating onset with *operando* impedance through an increase in graphite SEI resistance
- 3 electrode setup, large separator thickness results in Li plating at low SOCs





David Brown, Bryan McCloskey (Berkeley)

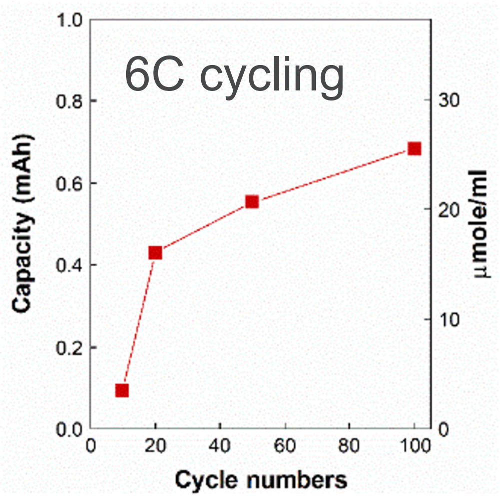
# Global Quantification of Li Plating

Goals:

- quantify the amount of Li
- detect onset of Li plating

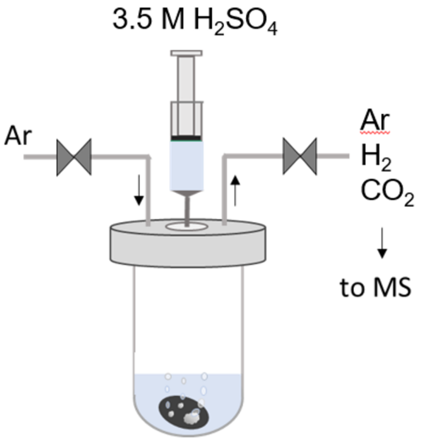
# Mass Spectrometry

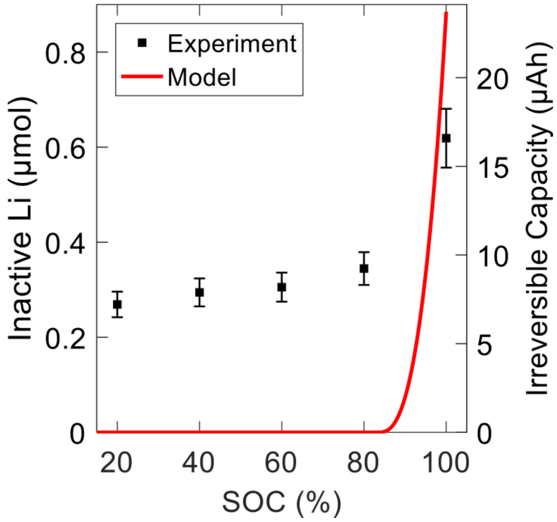
## Inductively coupled plasma mass spectrometry (ICP-MS)




- Quantify Li and F using ICP-MS
- Assume F is from LiF in SEI
- At 6C, amount of plated Li increases with increased cycle number

## Mass Spectrometry Titration (MST)



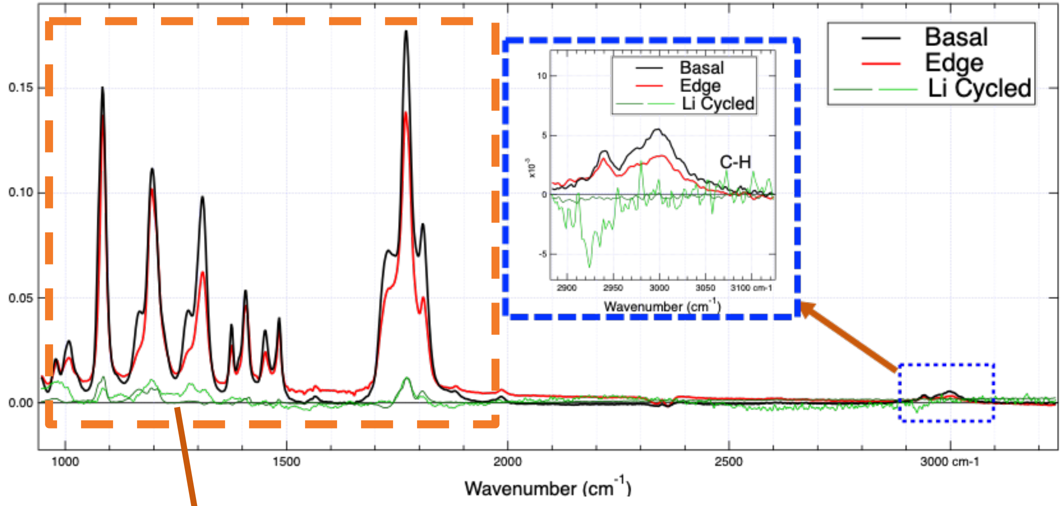

Plating onset at ~85%

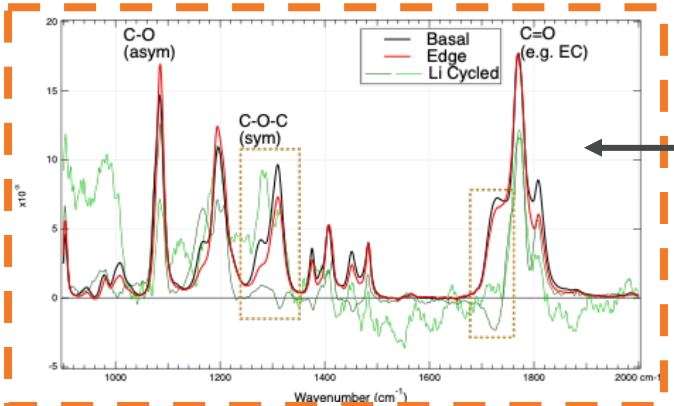
More detailed results in Poster BAT458

Experimental: Eric McShane, Bryan McCloskey (LBL)
Modeling: Andrew Colclasure, Kandler Smith (NREL)

# Fourier-transform infrared spectroscopy (FTIR)

- Detect Li plating by probing the SEI composition
- Comparing FTIR signal of SEI on graphite and on Li foil
- C-H absent on Li (blue box)
- Component ratio at ~1300 $cm^{-1}$ and C=O shoulder height at ~1730 $cm^{-1}$ differ (orange box)




Scaled to similar heights

Manuel Schnabel, Robert Kostecki (LBNL)

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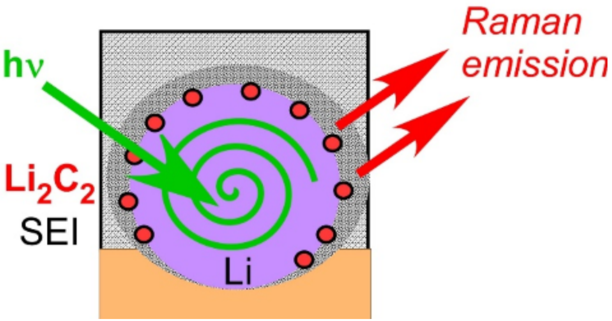
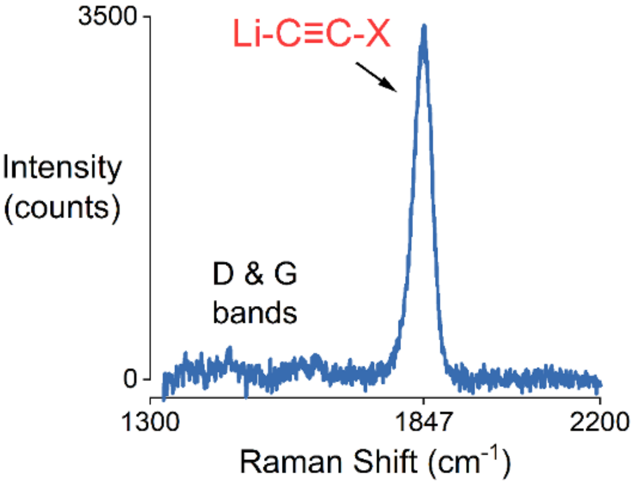
# Spatial Detection

Goal: identify *where* Li plates and connect to cell heterogeneity

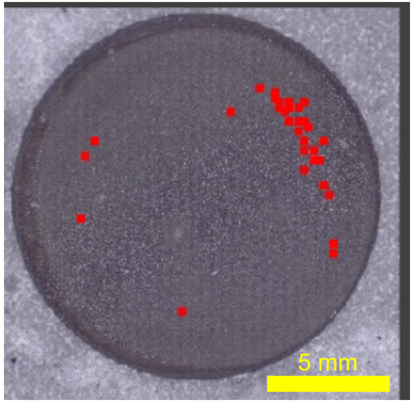
# Mapping Li Distribution with Raman Spectroscopy




Rodrigues et al., ACS Appl. Energy Mater. 2, 873 (2019)




Li plating map after **single** 6C CCCV charge



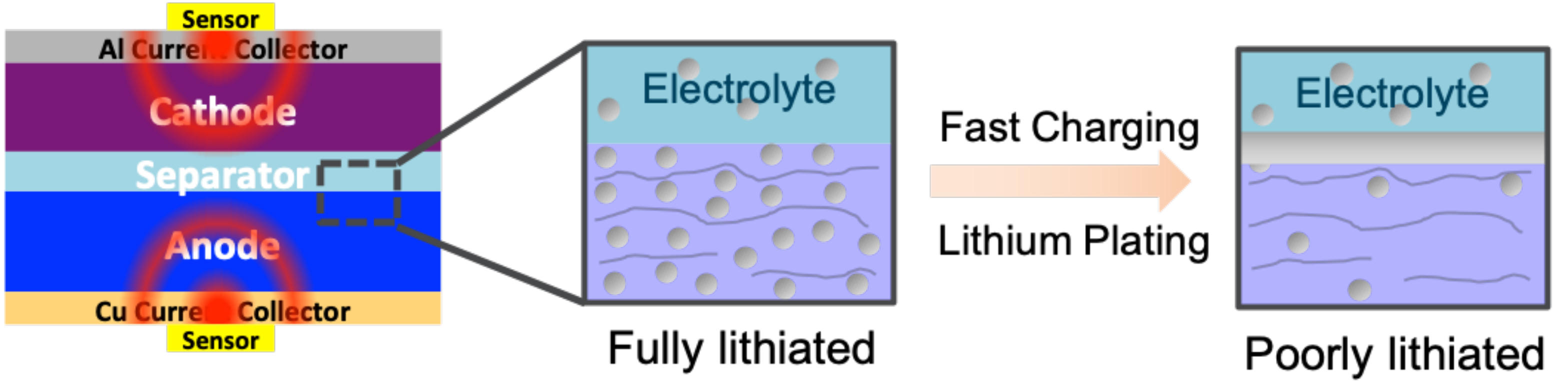

- Plated Li intensifies SEI bands through surface enhanced Raman spectroscopy-like mechanism
- Acetylide band used to determine Li presence

**Useful for studying early stages of Li plating during fast charge**




Marco Rodrigues Seoung-Bum Son, Abhi Raj, Ira Bloom, Daniel Abraham (ANL)

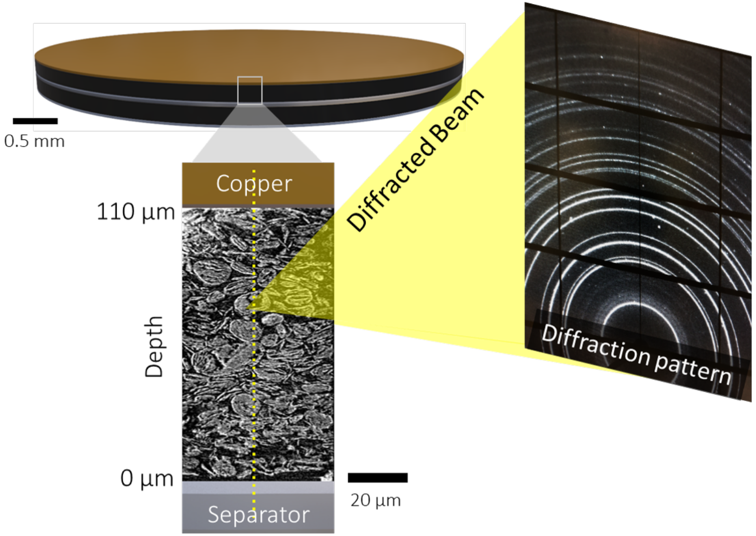
# Acoustic detection of Li plating with 3ω sensor




- Lithiation changes lattice constant & stiffness → affects thermal conductivity of electrodes
- Plated Li decreases interface thermal resistance between anode and separator
- Frequency dependence separates contribution from electrodes and interfaces

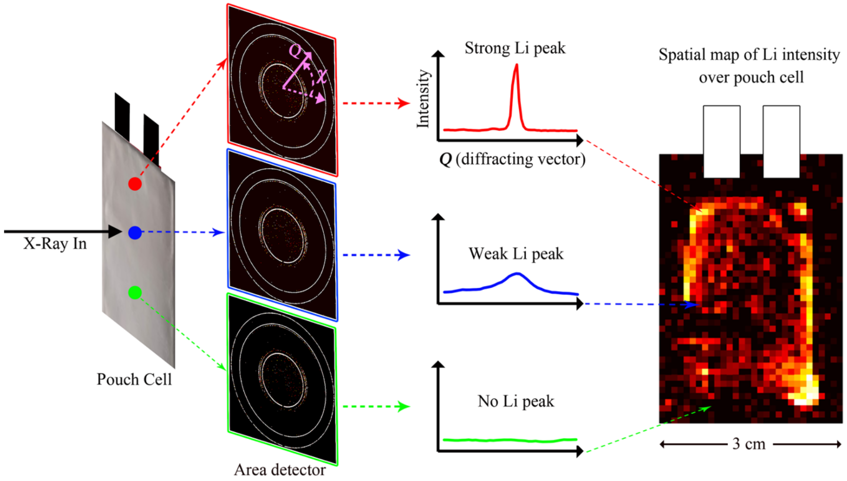
# Detecting Li with XRD mapping

## Li plating through anode thickness




Donel Finegan (NREL)

## Li plating across anode




Partha Paul, Chuntian Cao, Vivek Thampy, Hans-Georg Steinrück, Johanna Nelson Weker, Michael Toney (SLAC)

Locate and quantify where Li plating occurs in 3D space and correlate with other battery components (graphite stages, ….)

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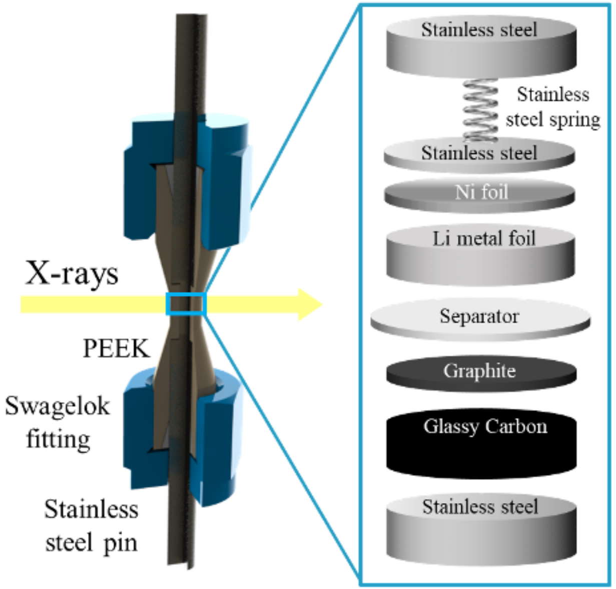
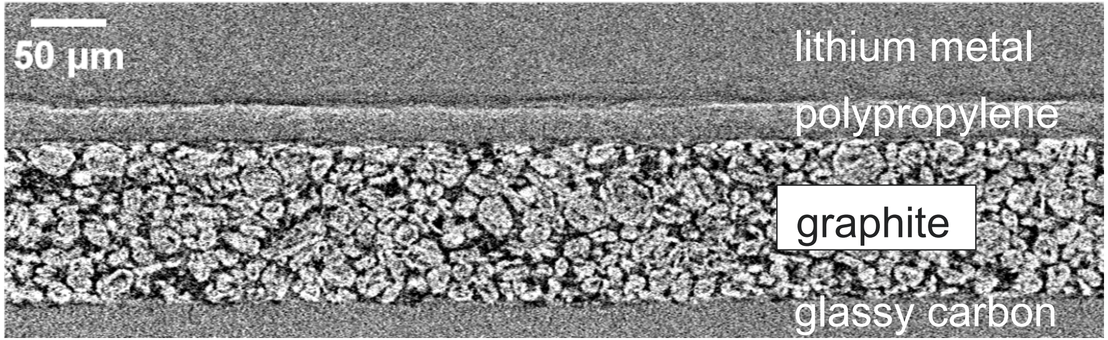
More detailed results in Poster BAT458

# Detecting Li with X-ray micron computed tomography (μCT) in Cu-free battery




✓ Cu free electrode enables high contrast, artifact-free μCT

Alec Ho, Donal Finegan, Wei Tong, Nitash Balsara (LBNL, NREL)

# Responses to previous year reviewers’ comments

## New Thrust Area for this project in response to reviewers’ comments

*Focus on the initial onset of plating rather than dramatic plating over the whole area*

- We are exploring techniques that are sensitive to detecting the onset of Li plating and investigating the detection limits for different techniques

*Split up the charging to everything that happens up to 50% and then what can happen after that*

- Many of techniques explore the onset of Li plate with SOC (dV/dt, MS)

# Remaining Challenges and Barriers

- Verify onset of detection
- Link Li plating onset with cell properties (local heterogeneities) and cycling performance
- Understand how Li plating evolve with aging

# Next steps

- Verify positive detection of Li onset with quantification techniques (such as MST)
- Connect capacity fade to amount of Li lost through irreversible plating (e.g. XRD mapping)
- Leverage *in situ* techniques (e.g. acoustic detection, XRD mapping, μCT) over the lifetime of the battery to understand the evolution Li plating

Any proposed future work is subject to change based on funding levels

# Summary

- Identify and contrast approaches to accurately detect and quantify Li plating during extreme fast charge conditions
- Link detection of onset of Li with local heterogeneity
- Combine techniques to answer when, where, and how Li plates
- Link detection of onset of Li with cell performance

# CONTRIBUTORS AND ACKNOWLEDGEMENTS

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